ECAR-3873 Acceptance of Nonconforming Fuel for the AGR-5/6/7 Irradiation Experiments

D. W. Marshall

September 2017



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Idaho National Laboratory Idaho Falls, Idaho 83415

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ENGINEERING CALCULATIONS AND ANALYSIS

INL/MIS-17-43272

Title: Acceptance of Nonconforming Fuel for the AGR-5/6/7 Irradiation Experiments

ECAR No.: 3873 Rev. No.: 0 Project No.: 23841 Date: 09/26/2017

SIGNATURES

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47	N/A			
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- 1. Confirmation of completeness, mathematical accuracy, and correctness of data and appropriateness of assumptions.
- 2. Concurrence of method or approach. See definition, LWP-10106.
- 3. Concurrence of procedure compliance. Concurrence with method/approach and conclusion.
- 4. Concurrence with the document's assumptions and input information. See definition of Acceptance, LWP-10200.
- 5. Does the document contain CUI material please check either yes or no.

TEM-10200-1 03/01/2012

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Rev. 06

Title: Acceptance of Nonconforming Fuel for the AGR-5/6/7 Irradiation Experiments

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REVISION LOG

Rev.	Date	Affected Pages	Revision Description
0	09/26/17	All	New document

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1.	Quality Level (QL) No.	QL-3	Professional Engineer's Stamp
2.	QL Determination No.	NA	N/A
3.	Engineering Job (EJ) No.	NA	See LWP-10010 for requirements.
4.	SSC ID	NA	
5.	Building NA	X	
6.	Site Area NA	X	

7. Objective/Purpose:

Document acceptance of graphitic fuel compacts fabricated for the Advanced Gas Reactor (AGR) irradiation experiments, AGR-5/6/7, despite non-conformance with four fuel specifications.

8. If revision, please state the reason and list sections and/or pages being affected:

9. Conclusions/Recommendations:

The fuel compacts, although not fully conforming to fuel specifications, are of sufficient quality that useful and meaningful data can be collected from the AGR-5/6/7 experiment irradiations. The fuel compacts are found to be acceptable for the purposes of the irradiations.

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PROJECT ROLES AND RESPONSIBILITIES

Project Role	Name (Typed)	Organization	Pages covered (if applicable)
Performer	D. W. Marshall	C620/C020	
Checker ^a	D. A. Petti	C004/C000	
Independent Reviewerb	N/A		
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Requestor ^e	P. A. Demkowicz	C600/C020	
Nuclear Safety ^e	N/A		
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Responsibilities:

- a. Confirmation of completeness, mathematical accuracy, and correctness of data and appropriateness of assumptions.
- b. Concurrence of method or approach. See definition, LWP-10106.
- c. Concurrence with the document's markings in accordance with LWP-11202.
- d. Concurrence of procedure compliance. Concurrence with method/approach and conclusion.
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NOTE:

Delete or mark "N/A" for project roles not engaged. Include ALL personnel and their roles listed above in the eCR system. The list of the roles above is not all inclusive. If needed, the list can be extended or reduced.

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SCOPE AND BRIEF DESCRIPTION

Prismatic Very-High Temperature Reactor (VHTR) fuel fabricated for the Advanced Gas Reactor (AGR) irradiation experiments, AGR-5/6/7, did not meet fuel specifications for the outer pyrocarbon (OPyC) mean thickness or the dispersed uranium fraction (DUF). Fuel was fabricated at two nominal packing fractions, 25% and 40%. In addition to the nonconforming properties already mentioned, the compacts with 25% packing fraction (PF) were nonconforming for the silicon carbide defect fraction and the compacts with 40% PF were nonconforming for the exposed kernel fraction (EKF).

Furthermore, because the impurities outside of the silicon carbide (SiC) layer all measured below the detection limit for the method, on a clutch of five compacts, the results were reported with units "µg/5-compact clutch" and could not be reported with the required units of "µg/compact."

This document describes the reasons why the nonconforming fuel compacts are deemed acceptable to use in the AGR-5/6/7 irradiation experiment.

DESIGN OR TECHNICAL PARAMETER INPUT AND SOURCES

None

BACKGROUND DATA

Property	Mean (N) ^a	Mean at 95% Confidence	Dispersion 0.95/0.99				
OPyC Thickness Specification ¹		36 - 44 μm	≤ 20 µm ^b				
TRISO lot J52R-16-98005 ²	35.03 μm	34.75 μm (F)	30.76 μm				
Dispersed U Fraction Specification ¹		≤ 1.0e-5					
TRISO lot J52R-16-98005 ²	2.28e-5						
40% PF Compacts 3, 4	3.18e-5	3.80e-5 (F)					
25% PF Compacts 3, 4	2.66e-5	2.95e-5 (F)					
Exposed Kernel Fraction Specification ¹		≤ 5.0e-5					
TRISO lot J52R-16-98005 ²	6.96e-6	9.35e-6 (N)					
40% PF Compacts 3, 4	6.57e-5	9.28e-5 (F)					
25% PF Compacts 3, 4	7.39e-6	1.48e-5					
SiC Defect Fraction Specification ¹		≤ 1.0e-4					
TRISO lot J52R-16-98005 ²	2.83e-5	3.27e-5 (N)					
40% PF Compacts ^{3, 4}	6.96e-5	9.66e-5					
25% PF Compacts ^{3, 4}	9.24e-5	1.22e-4 (F)					
a. (N) Property calculated, but not specified, and (F) fails to conform to the fuel specification.							

b. Not more than 1% of the population may be less than 20 μm at 95% statistical confidence.

ASSUMPTIONS

Not Applicable

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COMPUTER CODE VALIDATION

Not Applicable

DISCUSSION/ANALYSIS

TRISO OPyC Thickness

The OPyC layer performs multiple functions. 1) It is to serve as a final barrier to fission product releases, specifically for gaseous fission products. 2) It is known to shrink under fast neutron irradiation and presumed to compressively load the silicon carbide layer; thereby preventing the silicon carbide layer from failing due to hoop stresses caused by internal pressurization (a.k.a. pressure vessel failures; predominately from carbon monoxide accumulation). 3) The OPyC layer provides mechanical protection of the brittle SiC layer during subsequent handling.

The United States Advanced Gas Reactor Program will use a mixed ceramic fuel kernel that is composed of uranium carbides and oxides (UCO). UCO kernels evolve far less CO than urania kernels employed by other countries. Consequently, the internal pressures formed during irradiation are far less and hoop stress failures are effectively prevented. Furthermore, post-irradiation-examinations (PIE) on AGR-1 and AGR-2 fuels show a tendency for the OPyC to shrink away from the SiC vs. towards the SiC as previously imagined. Therefore, the OPyC layer does not impose a compressive load on the SiC as thought.

The fuel specification 1 for the OPyC thickness was based on all three functions, collectively. A major driver for the specification was adequate thickness to enable the OPyC to compress the SiC layer without cracking. Since the OPyC layer does not compress the SiC, as envisioned, and because thinner OPyC layers still adequately perform the functions of retaining gaseous fission products and protecting the SiC layer from mechanical damage, an OPyC thickness of $\sim 35~\mu m$ will be sufficient and no degradation of the TRISO in-pile performance is expected due to the mean thickness being slightly below the specification at 95% confidence.

The narrowly distributed OPyC thicknesses keep the lower tail of the distribution well above the critical limit of 20 μ m, which further supports the argument that the thickness will be adequate for the functions that it performs.

The AGR Technical Coordination Team (TCT) was consulted ⁵ before the TRISO batches were selected for composing the TRISO lot, knowing that conformance to the OPyC thickness specification could be jeopardized by the selection of batches. The TCT recommendation was: "BWXT TRISO coating batches 93165, 93168 and 93169 should be included in the coated particle composite to be used for AGR-5/6/7 compact formation," with full knowledge that inclusion of TRISO batch J52O-16-93165 would result in the mean OPyC thickness failing the fuel specification.

INL instructed BWXT to compose the TRISO lot from TRISO batches J52O-16-93165, 93168, 93169, and 93170 ⁶. BWXT issued a Quality Control Deficiency Notice (QCDN) ⁷ documenting that the composited TRISO lot did not meet the mean OPyC specification. The QCDN was accepted by INL with the disposition of "Approve as is."

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Compact Defect Fractions

The three defects that are quantified by the compact deconsolidation-leach-burn-leach (DLBL) method are the dispersed uranium fraction (DUF), exposed kernel fraction (EKF), and the silicon carbide defect fraction. The total contribution of all defects (DUF, EKF, and porous SiC) is to be kept below an effective 2e-4 defect level in a VHTR reactor to enable maintenance of heat transfer units and to prevent excessive off-site releases during accident events. The total allowable defect level is subdivided between the three defect fractions. Fission product releases from DUF is thought to be an order of magnitude higher, for a given mass of uranium, than releases from dense kernels with either cracked layers or porous SiC, thus the allowable defect level for DUF has been assigned a lower limit than the other two fractions.

Particulars of the three defect fractions are discussed individually below. The levels of the defects, which are higher than intended, were evaluated by INL and the TCT prior to accepting the fuel for use for the AGR-5/6/7 experiment irradiation ⁸. The reasons for accepting the fuel as-is include: 1) The fuel specifications were written for fuel to be used in a future VHTR reactor and not specifically written to safeguard data collection for an experiment irradiation. 2) The defect levels are high enough to complicate analysis of fuel performance data, but are not so high as to preclude "seeing" in-pile TRISO particle failures and collecting valuable data from the irradiation of the fuel. 3) Commercial VHTR fuel would need to undergo a "proof" test and not rely solely on AGR-5/6/7 data. 4) Refabricating the fuel would guarantee improved attributes. The required time to investigate the cause(s) of increasing defect levels during overcoating and compaction, in addition to that needed for fuel refabrication, would likely result in lost access to the Advanced Test Reactor Northeast flux trap, which is essential for irradiating a test train of the size designed for AGR-5/6/7.

Samples of overcoated TRISO particles for 40% PF compacts and compacts of both packing fractions are being analyzed at Oak Ridge National Laboratory for the three defect fractions to confirm the compact defect data obtained by BWX Technologies and to get a metric of damage done during deposition of the resinated graphite overcoat on the TRISO. The data are not available at this time.

Dispersed Uranium Fraction

Dispersed uranium fraction (DUF) is a variable property used to estimate the quantity of uranium outside of the SiC coating layer in the TRISO and compact. It is postulated that uranium can be incorporated into the OPyC layer from contamination on the coater walls or associated with the resinated graphite matrix. Impurities analyses of the components for the resinated graphite matrix preclude incorporation of significant natural uranium, leaving uranium contamination as the only true source.

DUF is quantified by assuming any quantity of uranium leached from the liberated TRISO particles amounting to less than one-half kernel equivalent, in the deconsolidating acid or in the pre-burn and post-burn leach sequences, is from contamination and that anything greater than this is from exposed kernels in cracked/broken particles or from porous silicon carbide layers. An intact OPyC layer is impervious to the acid used to deconsolidate the compact or leach prior to burning back the OPyC, so not much more than surface contamination should be detected with the pre-burn leaches. Burning back the OPyC liberates the remainder of the uranium embedded within the OPyC and making it accessible to the post-burn leach acids. The DUF is reported as the sum of the uranium recovered from all leaches combined, that are not attributed to either broken or porous coating layers.

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Leach-burn-leach tests on the TRISO lot indicated that the compacts would likely fail the DUF defect fraction. Because the estimated EKF and SiC defects for the TRISO lot would pass the compact specification, the TRISO was deemed acceptable. If the metric for DUF is accurate, then these defects should be unaffected during fuel compact formation. Comparison of the DUF calculated for the TRISO lot and the two compact packing fractions shows some increase in the DUF value; 25% PF increasing by $\sim 16\%$ and 40% PF by $\sim 39\%$.

Exposed Kernel Fraction

The exposed kernel fraction (EKF) is an attribute property estimating the fraction of damaged TRISO particles with cracked or broken coating layers such that the kernel is, at least, partially exposed. Even severely cracked coating layers and the fuel kernel, itself, retard the release of some fission products better than dispersed uranium. The TRISO particles are thought to incur damage by vacuum unloading of the TRISO (possibly only as incipient damage), during overcoating of particles with the resinated graphite matrix, and during fuel compact formation. The latter being the most significant source of damage or the process that exacerbates the incipient damage incurred in earlier processing steps.

TRISO lot data indicated that the EKF defect level was about 20% of the specification limit with a 95% confidence, suggesting that compacted fuel would conform to the specification. Some small increase in the EKF is suggested by the data for the 25% PF compacts, but it easily passed the specification requirement. The EKF for the 40% PF compacts, however, were an order of magnitude higher than the TRISO lot and fail at nearly double the specification limit.

Capsule 1 at the bottom of the test train and Capsule 5 at the top of the test train have compacts with 40% PF, collectively holding 114 fuel compacts. Capsules 2, 3, and 4 hold, collectively, 80 compacts at 25% PF. Irradiation of the 25% PF compacts is not impacted by high EKF of the 40% PF compacts. Capsule 1, with 90 compacts, is the most affected by the high EKF value. An assessment of the defect levels in Capsule 1 indicates that in-pile particle failures would still be detectable and that valuable data can be obtained during the irradiation and in post-irradiation examination (PIE), despite the high EKF value.

The calculated EKF mean and 95% confidence intervals, reported in the BWX Technologies compact certification package, were adjusted to account for the contribution of DUF in the pre-burn leaches. 4

Silicon Carbide Defect Fraction

The silicon carbide defect fraction is an attribute property estimating the fraction of particles with a porous or permeable SiC layer. Even porous SiC layers retain some fission products. Half of the allowable defect fraction for the reactor core is allocated to this SiC defect. This defect should be independent of the EKF, except as a weak spot where a fracture could initiate during fuel compact forming. The fraction of truly porous SiC layers should not increase during overcoating, compaction, or thermal treatment. Data show, however, that the mean SiC defect fraction increased to 330% and 250% of the TRISO defect level for the 25% PF and 40% PF compacts, respectively. This phenomenon is yet to be explained, but is probably due to cracked particle layers that manifested as porous SiC during the leaches by not allowing for complete leaching of the kernel. Nonetheless, the defects are attributed (by the method) to a SiC defect. The 25% PF compacts fail to conform to the specification while the 40% PF compacts barely pass.

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Compact Impurities Units of Measure

The fuel specification ¹ and the sampling plan ⁹ state that the units for impurities in the compacts be micrograms of the metal impurity outside of the SiC layer per compact. These analyses are performed on leachates generated during the compact deconsolidation-leach-burn-leach procedure for quantifying the DUF, EKF, and SiC defect fractions. A five-compact "clutch" is deconsolidated and leached in this method, so the measured impurities apply to the clutch and not individual compacts. All clutches measured impurities below the established detection limits for all elements of concern. BWX Technologies wrote: ¹⁰

"If the result reported by the analysis was greater than the LDL, then dividing the result by five would produce the desired unit. However, for cases where the reported result was "less than LDL", then dividing the LDL value by five would not be appropriate. That is because the LDL had been determined by the reproducibility of the ppb [parts per billion] calibration curve, which was independent of the number of compacts under test. Therefore, for those samples with elemental results reported at the LDL value, the minimum division possible is the clutch. For those situations, the desired unit of µg/compact cannot be achieved."

INL accepts reporting the compact clutch results, in lieu of compact averages, for this case. The maximum possible impurity per compact is, arguably, no more than the values reported for the clutch, and thereby pass the specification requirement with adequate assurance.

CONCLUSIONS

The consensus is that the OPyC thickness being below the specified range for the mean will not significantly contribute to in-pile particle failures, because the distribution is narrow and sufficiently high that the probability of having particles with less than a 20 µm thickness is very, very remote. Additionally, the specification for OPyC thickness was partially based on the assumption that shrinking OPyC would compress the SiC layer. Some mounted particles of irradiated fuel from the AGR-1 and AGR-2 fuels show that some OPyC layers shrink radially outward and debond from the SiC layer.

The high DUF and the higher than expected EKF and SiC defects is undesirable and likely would not be accepted use in a VHTR reactor. The defect levels will make detecting in-pile particle failures more difficult, but the judgement of subject matter experts is that our instrumentation will be able to discern the few particle failures that will occur and that valuable data will be obtained from the irradiation of the existing AGR-5/6/7 fuel compacts.

Despite the nonconforming properties of the TRISO and the compacted fuel, it is better to accept the fuel, as-is, than to attempt refabricating the fuel (with no guarantee of improvement) and risk losing access to the ATR Northeast flux trap for the irradiation.

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REFERENCES

1. SPC-3652, "AGR-5/6/7 Fuel Specification," Rev 8, March 2017.

- 2. BWXT Advanced Gas Reactor Program (AGR) Contract No. 107790, J52R, Industrial Fuel Fabrication and Development, Lot J52R-16-98005, Book 1 (BWXT N-74 Document Transmittal Form, "Revised Statistical Report on AGR 98005 Blend," Signed June 13, 2017.
- 3. BWXT Advanced Gas Reactor Program (AGR) Contract No. 107790, J52R, Industrial Fuel Fabrication and Development, LEU Compact Certification Package, Book 1.
- 4. BWXT N-74 Document Transmittal Form, "Supplemental Information to the AGR 5/6/7 Compact Certification Package," signed September 6, 2017, Re: DUF, EKF, and SiC defect calculations (Appendix A).
- 5. Technical Coordination Team February 1, 2017 teleconference summary issued February 8, 2017.
- 6. BWXT N-74 Document Transmittal Form, "Permission to Blend Mixture to Supply Material for AGR Compacting," signed February 20, 2017 (Appendix B).
- 7. BWXT N-74 Document Transmittal Form, "QCDN for missing OPyC thickness," signed March 21, 2017 (Appendix C).
- 8. Technical Coordination Team June 5, 2017 teleconference summary issued June 8, 2017.
- 9. PLN-4352, "Statistical Sampling Plan for AGR-5/6/7 Fuel Materials," Rev. 5, May 2016.
- 10. BWXT N-74 Document Transmittal Form, "Supplemental Information to the AGR 5/6/7 Compact Certification Package," signed September 12, 2017, Re: Impurities analyses and reported units (Appendix D).

APPENDICES

- A. BWXT N-74 Document Transmittal Form, "Supplemental Information to the AGR 5/6/7 Compact Certification Package," signed September 6, 2017, Re: DUF, EKF, and SiC defect calculations.
- B. BWXT N-74 Document Transmittal Form, "Permission to Blend Mixture to Supply Material for AGR Compacting," signed February 20, 2017.
- C. BWXT N-74 Document Transmittal Form, "QCDN for missing OPyC thickness," signed March 21, 2017.
- D. BWXT N-74 Document Transmittal Form, "Supplemental Information to the AGR 5/6/7 Compact Certification Package," signed September 12, 2017, Re: Impurities analyses and reported units.

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Appendix A

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BWXT RTRT

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DOCUMENT TRANSMITTAL FORM

N-74 Rev. 2 (02/26/16) (CR-1044961-01) Page 1 of 1

CUSTOMER 1077 CONTRACT:	PROJECT		nced Gas Reactor BWXT J52R PAC / NPN NO. J52R
	James and American to the American	the ACD	R 5/6/7 Compact Certification Package
DESCRIPTION: Supp	lemental information to	the AGK	
BWXT DOCUMENT NUMBER:	N/A		REVISION:
(Note to CDC: when FAX'd or	DATE: r delivered to BWXT mail room)	/	/ TIME/# Pgs: 2.05 3
		/2017	☐ FOR INFORMATION
TO: Idaho National Laboratory P. O. Box 1625 Idaho Falls, ID 83415 phone number 208-526-2930 fax number 208-526-2930	357	FROM: (see originator's name, phone, mail code below)	BWXT NOG-L USPS P.O. Box 785 Lynchburg, VA, USA 24505-0785 courier Mt. Athos Road Lynchburg, VA, USA 24504 FAX 434/522-5410
ATTN: Doug Marshall			
The attached memorandum that BWXT used to charact BWXT ORIGINATOR: NAME: Alex Tilton BWXT MANAGEMENT: NAME: Dave Navolio	EXT / MAIL . CODE: (5394/ 061	SIGNATURE / DATE: SIGNATURE / DATE: 9/6/17 SIGNATURE / DATE:
		061	N. M. 196/11
CUSTOMER DISPOSITION ACCEPTED COMMENTS:	ON: (customer may substitute equ		
NAME: Douglas W.	Marshall EXTI		SIGNATURE / DATE: No Marshall 9/6/17

ADMINISTRATIVE REVIEW:

ENGINEERING CALCULATIONS AND ANALYSIS

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As part of the certification of the AGR 5/6/7 LEU Compacts, the fraction of the TRISO particles inside each compact was characterized. INL specifications PLN-4352 and SPC-1352 documented the testing requirements and the corresponding limits for each test. A 95% confidence interval was specified for the failure fraction attributes. Once the raw data had been obtained from the BWXT Chemistry Laboratory, the content was forwarded to the BWXT Statistics Department for statistical characterization. The content below summarizes the failure fraction testing method and the statistical method that was used for the characterization.

For both the 40% PF and the 25% PF cases, the results from the Chern Lab were reported as equivalent particle failures by 5-compact clutch for each leach operation. The leaches were divided between pre-burn and post burn. All clutches received at least one leach operation (pre-burn and post-burn), with some of the clutches receiving three. The results from the leaches were summed into a total before burn and a total after burn. Those clutches where the sum was less than 0.5 equivalent particles were grouped into the DUF attribute and were not considered for the EKF or SIC calculations. Once the DUF had been determined, the result was subtracted from each clutch total. The pre-burn DUF was subtracted from the pre-burn clutch result, and the post-burn DUF was subtracted from the post-burn clutch result. For the EKF attribute, the pre-burn differences were summed, rounded, and divided by the number of particles present in all the clutches. For the SIC attribute, the post-burn differences were summed, rounded, and divided by the number of particles in all the clutches. The result was a total equivalent particle failure fraction representing the pre-burn leaches (EKF) and the post-burn leaches (SIC). This information, along with the raw data, was provided to the BWXT Stats group for analysis.

The analysis began with a review of the Program specifications. PLN-4352 and SPC-1352 were reviewed for the contractual requirements. It was decided that the data would best be represented by a Poisson Distribution. The probability distribution of the Poisson random variable X, representing the number of failed particles in a given number of particles is

$$p(x;u) = \frac{e^{-u}u^x}{x!}$$

where u is the average number of failures occurring and e=2.71828. The probability distribution is calculated for each interval from 0 on, until the cumulative probability hits .95, at which point we can say that we are 95% confident that the number of particles is less than or equal to the number corresponding to the 95% cumulative probability.

An example of what this distribution looks like is shown in Figure 1.

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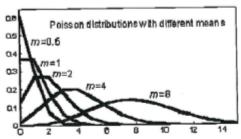


Figure 1: Example of Poisson Distributions

For the case of the 40% PF EKF, the 50th percentile of the curve was centered at the number of pre-burn equivalent particles, or 17. For that curve, the point that represents 95% of the area under the curve would be 24. Therefore, there is a 95% probability that the mean of the distribution is less than or equal to 24. A similar process was used for the 40% PF SIC attribute and both the 25% PF attributes. Once the equivalent particle failures had been determined, each value was divided by the total number of particles tested to generate the failure fraction.

Below is the table containing the failure fraction reported values.

Packing Fraction	Attribute	Mean Result	95% Confidence	Spec	Result
40%	EKF	6.57E-05		≤5.0E-05	FAIL
40%	SIC	6.96E-05	9.66E-05	≤1.0E-04	PASS
25%	EKF	7.39E-06	1.488-05	≤5.0E-05	PASS
25%	SIC	9.24E-05	1.22E-04	≤1.0E-04	FAIL

Table 1: Fallure Fraction Data Summary

TEM-10200-1 03/01/2012

Rev. 06

ENGINEERING CALCULATIONS AND ANALYSIS

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Title: Acceptance of Nonconforming Fuel for the AGR-5/6/7 Irradiation Experiments

ECAR No.: 3873 Rev. No.: 0 Project No.: 23841 Date: 09/26/2017

Appendix B

ENGINEERING CALCULATIONS AND ANALYSIS

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Title:

Acceptance of Nonconforming Fuel for the AGR-5/6/7 Irradiation Experiments

ECAR No.: 3873 Rev. No.: 0 Project No.: 23841 Date: 09/26/2017



DOCUMENT TRANSMITTAL FORM

N-74 Rev. 2 (92/26/16) (CR-1044961-01) Page 1 of 1

CUSTOMER CONTRACT	107790	PROJECT		ed Gas F	PAC / NE	N NO. J52	R
DESCRIPTION:	Permission to BI	end Mixture to Su				g	
BWXT DOCUMEN	T NUMBER: NA				REVISION:	00	
	C: when FAX d or poliver	DATE;	2 / 20	/17	TIME /# Pgs:		3
	ROVAL - NEED		17		☐ FOR	INFORMA	TION-
address address city, stat phone n	e, country, zip: Idaho Falls umber: 208-526-3857 per: 208-526-2930		FROM: (see organistors name phone imali score bottow)	USPS (BWXT NOG-L P.O. Box 785 -ynchburg, VA, USA : ML Athos Road Lynchburg, VA, USA 134/522-5410		
1 1 2 2 3 1 2	Marshall						
BWXT ORIGIN	as part of the TR	year years	oe used for 3177 / 61		URE / DATE: /	2-20-1 2	1. 4. 1. 1.
BWXT MANAC		CODE	450/61	SIGNAT	URE / BATE:	2/20/17	1:5
CUSTOMER	ISPOSITION: (cut	domer may substitute oqu	ivalent form)	a majerina Geografia	CHIEF PER	15 A SEC.	i kar
ACC	EPTED [ACCEPTED	AS NOTED		□ NOT AC	CEPTED	
COMMENTS:							
NAME:	uglas W. Mar	shall MAIL CODE:		SIGNAT	TURE / DATE:	2/201	zoir

ENGINEERING CALCULATIONS AND ANALYSIS

Page B3 of B4

Title:

Acceptance of Nonconforming Fuel for the AGR-5/6/7 Irradiation Experiments

ECAR No.: 3873

Rev. No.: 0

Project No.:

23841

Date: 09/26/2017

Keeley, Joseph T (Joe)

From:

Douglas Marshall < opamarshall@gmail.com>

Sent:

Friday, February 17, 2017 10:36 AM

To: Cc:

Keeley, Joseph T (Joe); Marshall, Douglas W Navolio, David W; Niedzialek, Scott E; Richardson, W C (Clay); Jones, Aaron C; Mulreany,

Robert E

Subject:

EXTERNAL:Re: FW: AGR Burn Leach results

Joe, et al.,

The data look far better than I had hoped for. Both leaches pass the compact specifications at 95% confidence. We saw, as you stated earlier, more than 50% reduction of the pre-burn leach and nearly 75% reduction in the post-burn leach calculated at 95% confidences.

		Pri	e Burn Leach	Post Burn Leach				
			Total				Total	
	Failed Part	# Porticles	Failure Fraction	95%Conf.	Failed Part	# Particles	Fellure Fraction	95% Conf.
93165A	2.21	330000	6.70E-06	1.91E-05	2.78	165000	1.686-05	4.70E-05
93166RA	1.57	318000	4.94E-06	1.98E-05	0.92	159000	5.79E-06	2.98E-05
93168A	3.71	324000	1.15E-05	2.83E-05	2.27	162000	1.40E-05	3.896-05
93169A	10.37	324000	3.206-05	Market Street	2.86	162000	1.77E-05	4.79E-05
93170A	THE RESERVE OF	CONTROL OF THE PARTY.	2.15E-05	4.06E-05	Mark Mark		2.28E-04	T TO SE
93170A extra	2000		1.06E-04	CONTROL OF THE PARTY OF THE PAR			1.676-04	
2027qrt days	- Indiana		4.26E-05	art			2.00E-04	W. 12740 .
93170B	BURNOTH WAS	AND THE REAL PROPERTY.	1.06E-05	3,896-05	BM 1775		2,49E-05	5.65E-05
	100	10	1.485-05	3.89E-05	100	Table 1	6.22E-05	
981708 931708 total	W		1.27E-05	2.83E-85	I Control		4.35E-05	6.75E-45

Please use 93170 as part of the LEU TRISO lot blend.

Douglas Marshall

On Fri, Feb 17, 2017 at 7:18 AM, Keeley, Joseph T (Joe) < itkeeley@bwxt.com> wrote:

I made an error and corrected it in the table. I added the failure fractions and what I should have totaled all failed parts and divided by the total number of particles. The conclusion is still the same, we made significant improvements.

From: Keeley, Joseph T (Joe)

Sent: Friday, February 17, 2017 8:49 AM

To: 'opamarshall@gmail.com'

Cc: Navollo, David W; Niedzialek, Scott E; Richardson, W C (Clay); Jones, Aaron C; Mulreany, Robert E

Subject: AGR Burn Leach results

Doug,

ENGINEERING CALCULATIONS AND ANALYSIS

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Title:

Acceptance of Nonconforming Fuel for the AGR-5/6/7 Irradiation Experiments

ECAR No.: 3873 Rev. No.: 0 Project No.: 23841 Date: 09/26/2017

I've updated the results table with the burn leach results for 93170B. We made significant improvements to both the pre-leach and post-leach results and are now with specification. I know Dave set a teleconference for 11 am, but if the results look good to you and we can go ahead processing, please call me sooner (434-522-6177). I'd like to try and get our Operations group working on this as soon as possible.

Thanks,

Joe

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Title: Acceptance of Nonconforming Fuel for the AGR-5/6/7 Irradiation Experiments

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Appendix C

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Title:

Acceptance of Nonconforming Fuel for the AGR-5/6/7 Irradiation Experiments

ECAR No.: 3873 Rev. No.: 0 Project No.: 23841 Date: 09/26/2017

03/21/2017 08:10 FAA 434 522 5410

BAYL WIRT

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DOCUMENT TRANSMITTAL FORM

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CUSTOMER 107790	CUSTOME! PROJECT:	獻		PAC/N	PN NO. J52	R		
and the state of t								
	29 (19 29 4							
(Note to CDC; when FAX'd or delive	DATE:	2 / 20	/17	TIME / # Pgs:	7:55	2		
☑ FOR APPROVAL ~ NEED		7			INFORMA	TION		
TO: company name: Idaho National Leboratory address 1: PO Box 1625 address 2 city, state, country, zip: Idaho Fatis, ID 83415 phone number: 208-526-3857 phone number: 208-526-3857 fax number: 208-526-2930 in care of (c/o) facility of the number 208-526-2930 facility of the number: 208-526-2930 facility of								
ATTN: Doug Marshall								
Attached is the QCDN for missing the OPyC thickness on the AGR coating blend J52R-16-98005 (approved by INL on February 8, 2017). The violation was noted in the Met Lab samples that were split from Containers #1 and #2 of the blend. FYI, the weighted average I reported in the QCDN came from the batch weights used in the blend and the thickness data from the "A" material. We did not collect thickness data on the "B" material (after the resieve). If you have any questions please contact Dave or me. BWXT ORIGINATOR: SIGNATURE / DATE: 3 - 21 - 17								
DAVED NEVOLG	MAIL 64	[6]	NOW.	Ilan	المارار			
CUSTOMER DISPOSITION: (cu				1 100 - 100	denses	<u>! </u>		
	ACCEPTED A	SNOTED) ·	NOT ACC	CEPTED			
COMMENTS: Recent post-irradiation examination observations from previous AGR experiments suggest that the measured OPy C thickness on TRISO batches composing lot J5ZR-16-98005 will not have a deleterious effect on in-pile performance. J5ZR-16-9805 is accepted for use as is.								
NAME: Douglas Marsh	MAIL CODE:		SIGNATURE	eshall	3/21/1	7		

ADMINISTRATIVE REVIEW: Ke & Hartless 3/24/17

ENGINEERING CALCULATIONS AND ANALYSIS

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Title:

Acceptance of Nonconforming Fuel for the AGR-5/6/7 Irradiation Experiments

ECAR No.: 3873 Rev. No.: 0 Project No.: 23841 Date: 09/26/2017

03/21/2017 08:10 FAX 434 522 5410 RAYL KIKL @UU2 Page 1 of 1 Q11-127 Quality Control Deficiency Notice (U) Rev 01 March 11, 2016 CR-1044961-01 QCDN No. J52-004 Dept / Section UPRR QC Material ID: J52R-16-98005 Stage of Processing: Blend Contract No.: 107790 Contract Name: AGR Requirement Violated: OPyC thickness Data Attached YES Route Card Seq. No. Defect Description Actual Requirement N/A Sample Data 1 = 35.1 Sample Data 2 = 34.5 36 - 44 Correction Action Required Applicable Not Applicable X Responsible Area: Engineering Manufacturing___ QC___ Corrective Action Comments CA# N/A Corrective Action By______ Date_____ QA Engr._ Continue Process, No Further Evaluation Necessary Submit for Customer Disposition RC Step Continue Process, Evaluate at Repair / Rework Via RC_ Hold (By_ Engr. Comments / Actions: The blend was made from four batches (93165B, 3057.1 g, 30.3 µm; 93168B, 2995.8 g, 38.5 µm; 93169B, 2747.6 g, 36.0 µm an 93170B, 2845.4g, 35.6 µm). The thickness of 93165B and 93170B were low. The weighted average thickness was calculated to be 35.1 µm. Process Engr. Word Y. Keely Date 3/zo/17 M Uluy QA Engr. Customer Disposition: Approve as is______ Reject Approve with the following Conditions ___ Customer Signature New Marshell Date 3/21/2017 Process Engr._____ Date__ QA Engr._____ Date

ENGINEERING CALCULATIONS AND ANALYSIS

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Title: Acceptance of Nonconforming Fuel for the AGR-5/6/7 Irradiation Experiments

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Appendix D

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Title:

Acceptance of Nonconforming Fuel for the AGR-5/6/7 Irradiation Experiments

ECAR No.: 3873

Rev. No.: 0

Project No.:

23841

Date: 09/26/2017

09/11/2017 10:35 FAX 434 522 5410

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CUSTOMER CONTRACT:	1077	90 (0	ROJEC;			PAC / N	PN NO. J52R
·				APPROVAL do			
DESCRIPTION:	Supp	lemental Inf	ormation	to the AGR	5/6/7 C	ompact Certific	ation Package
BWXT DOCUMEN	IT NUMBER:	N/A				REVISION:	
(Note to CD	C: when FAX'd or	delivered to BWX	DATE; T mail room)		/2017	FIME / # Pgs: (FAX only)	10:20am 3
	ROVAL N	EED DATE	9/1	2/2017		☐ FOR	INFORMATION
TO: Idaho National Laboratory P. O. Box 1625 Idaho Falls, ID 83415 phone number 208-526-3657 fax number 208-525-2930 Idaho National Laboratory P. O. Box 1625 Idaho Falls, ID 83415 Idaho Fal							
ATTN: Doug	Marshall					-	
The attached memorandum clarifies BWXT's impurities testing approach. Please note that the outstanding statistical calculation for iron and transition metals will be reported as micrograms per clutch. These calculations will begin as soon as BWXT has INL approval to proceed. BWXT ORIGINATOR: NAME: Alex Tilton BWXT MANAGEMENT: NAME: Dave Navolio BWXT MANAGEMENT: NAME: Dave Navolio BWXT MANAGEMENT: NAME: Dave Navolio SIGNATURE / DATE: OCODE: 061 SIGNATURE / DATE: OCODE: 061							
Dave N	avolio		MAIL	6450/ 061	W.	Moshi "	7/11/2017
CUSTOMER D	SPOSITION	· /customer may	cuhaWuta co	uluslant form)			1
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COMMENTS:							
NAME: Dou	glas W. M	Mawhall	MAIL CODE:		SIGNATI	URE/DATE: aushall 9/	113/17

ADMINISTRATIVE REVIEW:

Kelly A. Hartless

ENGINEERING CALCULATIONS AND ANALYSIS

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Title: Acceptance of Nonconforming Fuel for the AGR-5/6/7 Irradiation Experiments

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Testing of the impurities present in the AGR 5/6/7 Compacts was specified in INL Documents SPC-1352 and PLN-4352. The limits for each metallic impurity are listed in the respective table in the two documents. To summarize the process that is used for the impurity testing, a set of randomly sampled compacts from each lot are selected. The number of compacts selected for each lot is determined by the number of TRISO particles per compact and the statistical confidence required for the reported results. Once the compacts have been removed from the lot, they are placed into one container and transferred to the BWXT Chemistry Laboratory. The compacts are randomly removed from the container into groups of five compacts, known as a clutch of compacts, or clutch. The compacts in each clutch are deconsolidated into TRISO particles and residual solids using an electrochemical process with nitric acid. Following deconsolidation, the solution is leached at just below the boiling point of the acid. The solids and the TRISO particles are separated from the solution and all the solids (including the TRISO particles) are burned to remove the excess carbon. Following the burn, the residual solids are again leached in hot nitric acid. Samples of the solution prior to the burn and post-burn are retained for impurity testing. The impurity testing is done using an ICP-MS (Inductively Coupled Plasma Mass Spectrometer). Each sample (pre-burn and post-burn by clutch) is first diluted using a dilution factor of 200 to minimize the corrosive effects of the acid inside the torch. The diluted sample is injected into the torch of the ICP-MS (Nu Instruments Attom HR ICP-MS, Figure 1) and the analyzer is tuned to the

specified elements. The mass spectrometer records the counts associated with each specified elements. The conversion of the counts to concentration is done using a parts-per-billion (ppb) calibration curve obtained using standards. The ppb concentration corresponding to the accumulated counts is further corrected for the dilution factor prior to being reported by the unit. A lower detection limit (LDL) for each element is established by the chemist based upon the precision and confidence found at the lower concentration levels found in the samples. If the elemental value for a given sample is reported to be less than the LDL, the LDL value is used. If the elemental value is greater than the LDL, then the reported value is used. Note that during the



Figure 1: Attom High Resolution ICP-MS

previous discussion, the sample under analysis was obtained from a clutch of five compacts. The requirement in the two INL documents referenced above was that the results be reported in micrograms per compact ($\mu g/compact$). If the result reported by the analysis was greater than the LDL, then dividing the result by five would produce the desired unit. However, for cases where the reported result was "less than LDL", then dividing the LDL value by five would not be appropriate. That is

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Title: Acceptance of Nonconforming Fuel for the AGR-5/6/7 Irradiation Experiments

ECAR No.: 3873 Rev. No.: 0 Project No.: 23841 Date: 09/26/2017



because the LDL had been determined by the reproducibility of the ppb calibration curve, which was independent of the number of compacts under test. Therefore, for those samples with elemental results reported at the LDL value, the minimum division possible is the clutch. For those situations, the desired unit of μ g/compact cannot be achieved.

Table 1 below demonstrates that the results for iron and the Transition Metals were all below the LDL for all samples. Note also in the table that the results are listed as $\mu g/clutch$. The results in the table will be forwarded to the BWXT Statistical Department for the remaining statistical calculations, as defined in the INL Specifications.

Lot	Sample #	MS Detection Limit (µg/clutch)					
		Fe	Со	Cr	Mn	Ni	Sum Co, Cr, Mn, Ni
J52R-16-14154C	1	<5	<10	<25	<10	<10	<55
J52R-16-14154C	2	<5	<10	<25	<10	<10	<55
J52R-16-14154C	3	<5	<10	<25	<10	<10	<55
J52R-16-14154C	4	<5	<10	<25	<10	<10	<55
J52R-16-14154C	5	<5	<10	<25	<10	<10	<55
J52R-16-14154C	6	<5	<10	<25	<10	<10	<55
J52R-16-14155C	1	<5	<10	<25	<10	<10	<55
J52R-16-14155C	2	<5	<10	<25	<10	<10	<55
J52R-16-14155C	3	<5	<10	<25	<10	<10	<55
J52R-16-14155C	4	<5	<10	<25	<10	<10	<55
J52R-16-14155C	5	<5	<10	<25	<10	<10	<55
J52R-16-14155C	6	<5	<10	<25	<10	<10	<55
J52R-16-14156C	1	<5	<10	<25	<10	<10	<55
J52R-16-14156C	2	<5	<10	<25	<10	<10	<55
J52R-16-14156C	3	<5	<10	<25	<10	<10	<55
J52R-16-14156C	4	<5	<10	<25	<10	<10	<55
J52R-16-14156C	5	<5	<10	<25	<10	<10	<55
J52R-16-14156C	6	<5	<10	<25	<10	<10	<55
J52R-16-14157C	1	<5	<10	<25	<10	<10	<55
J52R-16-14157C	2	<5	<10	<25	<10	<10	<55
J52R-16-14157C	3	<5	<10	<25	<10	<10	<55
J52R-16-141570	4	<5	<10	<25	<10	<10	<55
J52R-16-141570	5	<5	<10	<25	<10	<10	<55
J52R-16-141570	6	<5	<10	<25	<10	<10	<55

Table 1: Summary Impurity Data